

## Characterization of 3C-SiC Films Grown on 4H- and 6H-SiC Substrate Mesas During Step-Free Surface Heteroepitaxy

Philip G. Neudeck<sup>1</sup>, J. Anthony Powell<sup>1</sup>, David J. Spry<sup>2</sup>, Andrew J. Trunek<sup>2</sup>,  
Xianrong Huang<sup>3</sup>, William M. Vetter<sup>3</sup>, Michael Dudley<sup>3</sup>,  
Marek Skowronski<sup>4</sup> and Jinqiang Liu<sup>4</sup>

<sup>1</sup>NASA Glenn Research Center, 21000 Brookpark Road, M.S. 77-1, Cleveland, OH 44135 USA

<sup>2</sup>OAI, 21000 Brookpark Road, M.S. 77-1, Cleveland, OH 44135 USA

<sup>3</sup>Dept. of Materials Science & Engineering, SUNY Stony Brook, Stony Brook, NY 11794 USA

<sup>4</sup>Materials Science Dept., Carnegie Mellon University, Pittsburgh, PA 15213 USA

**Keywords:** 3C-SiC, cubic-SiC, epitaxial growth, heteroepitaxy, step-free surface heteroepitaxy

**Abstract.** This paper reports detailed structural characterization of 3C-SiC heteroepitaxial films grown on 4H- and 6H-SiC mesa surfaces. 3C-SiC heterofilms grown by the “step-free surface heteroepitaxy” process, free of double-positioning boundary (DPB) and stacking-fault (SF) defects, were compared to less-optimized 3C-SiC heterofilms using High Resolution X-ray Diffraction (HRXRD), High Resolution Cross-sectional Transmission Electron Microscopy (HRXTEM), molten potassium hydroxide (KOH) etching, and dry thermal oxidation. The results suggest that step free surface heteroepitaxy enables remarkably benign partial lattice mismatch strain relief during heterofilm growth.

### Introduction

The ability to reproducibly grow device-sized defect-free 3C-SiC crystals could enable new SiC devices to be realized, including improved SiC MOSFET devices [1] and heteropolytype junction devices [2]. However, the development of 3C-SiC electronic devices has been hindered by the fact that almost all 3C-SiC crystals, including those grown on undulant silicon, contain planar structural defects [3].

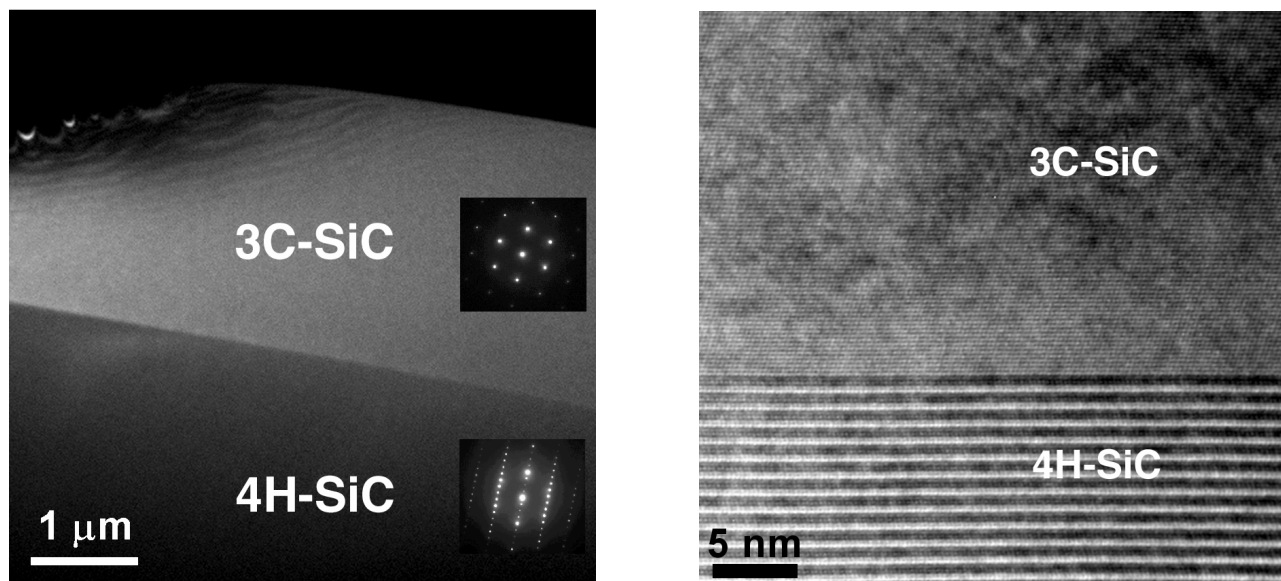
At ICSCRM-2001, we introduced a “step-free surface heteroepitaxy” growth process that achieved 3C-SiC films that appear to be completely free of double positioning boundary (DBP) and stacking fault (SF) planar defects [4]. The 3C films were grown on top of 4H- and 6H-SiC mesas (up to 0.4 mm x 0.4 mm) etched into commercial on-axis substrates. In this process, screw-dislocation free substrate mesas are first rendered step-free by performing homoepitaxial growth under pure stepflow conditions [5]. Then, by lowering the growth temperature in-situ, two-dimensional (2D) nucleation was induced on the step-free (0001) 4H or 6H mesa basal plane. Heteroepitaxial growth of 3C-SiC becomes thermodynamically favorable in these growth conditions without kinetic “step-control” to replicate substrate polytype [6]. Nucleation of 3C-SiC on step-free mesas successfully eliminated DPB defects, indicating (for the growth conditions studied) that the local stacking sequence of the top two substrate bilayers thermodynamically controls which twin-variant of 3C-SiC nucleates on a 4H- or 6H-SiC (0001) basal plane [4,7,8].

The step-free surface heteroepitaxy process is also based upon low (but nonzero) nucleation rates for the initial stages of heterofilm growth. In particular, 3C films initiated on step-free mesas at higher nucleation rates yielded 3C-SiC films with numerous SF defects, while 3C films initiated at lower nucleation rates (i.e., grown by step-free surface heteroepitaxy) were free of SF defects [4]. The observed behavior of 3C film quality as a function of growth initiation process has led us

(Neudeck and Powell) to hypothesize that stacking faults arise in this process when multiple 3C islands 2D nucleate on a single mesa, laterally expand across the step-free hexagonal-SiC growth surface, and coalesce in a defective manner [4]. In particular, we propose that strain and/or strain relief effects arising from in-plane lattice mismatch between the 3C-SiC film and 4H/6H mesa causes defective island coalescence leading to SF defect formation. However, more complete experimental data is necessary to fully substantiate the proposed growth and defect formation model. Previous characterization of 3C-SiC heterofilms grown on step-free 4H/6H mesas primarily relied upon thermal oxidation and X-ray topography to map crystal polytype and extended crystal defects [4,8]. This study attempts to more fully elucidate important structural properties of the 3C-SiC heterofilms (such as in-plane mesa/film lattice mismatch) grown on step-free mesa surfaces by applying additional characterization techniques.

### HRXTEM

Thermal oxidation defect mapping reveals stacking fault defects that propagate along three of the four equivalent  $\langle 111 \rangle$  planes of 3C-SiC, as these three planes intersect the top surface of the film where the enhanced surface oxide growth along the fault can be readily observed [9]. However, stacking disorder of a few of bilayers running parallel to the 3C/4H (or 3C/6H) heterointerface would not be observable by either oxidation or X-ray topography. Under previous growth models [6], such stacking disorder might be expected when film growth takes place (as in this experiment) via 2D terrace nucleation. A SF-free 3C film grown on a 4H-SiC mesa from Sample C described in [4] was selected for study by HRXTEM. The HRXTEM data shown in Fig. 1 indicates a structurally perfect 3C-SiC film with no defects and no stacking disorder detected within the field of view. The 3C/4H interface was perfectly flat and atomically abrupt with no evidence of growth steps and/or dislocations. Other regions studied produced similar findings. Another mesa selected for study was revealed by thermal oxidation to contain a single stacking fault defect. HRXTEM revealed the fault at low magnification, while high magnification analysis showed the SF to be the expected misalignment of atoms across a single  $\langle 111 \rangle$  plane that penetrated all but a few bilayers of the 3C film [10].



**Fig. 1:** HRXTEM of SF-free 3C-SiC heterofilm on 4H-SiC mesa (left) at low magnification and (right) high magnification. No defects and no stacking disorder was observed in the 3C heterofilm.

## HRXRD

Given the SF defect formation model discussed in the introduction, it is crucial to definitively ascertain in-plane lattice mismatch between the 3C films and the 4H (or 6H) mesa substrates. Therefore, a detailed HRXRD study was undertaken to ascertain lattice mismatch and film stress relaxation present in these heterofilms.

The HRXRD measurements of both mismatch and absolute lattice constants, too extensive to be fully detailed within this paper, are discussed elsewhere [11,12]. Three summary observations are directly relevant to the epitaxial growth discussion of this article: 1) In-plane substrate/epilayer lattice constant mismatch ( $\Delta a/a$  range of 0.02% to 0.09%) was observed on all samples indicating that some lattice mismatch strain relief occurred in the 3C film. Meanwhile, the measured out-of-plane lattice constant difference  $\Delta c/c$  was  $-0.13\%$  to  $-0.15\%$  for 3C on 4H and around  $-0.092\%$  for 3C on 6H. 2) The 3C-SiC films are not fully relaxed, as the measured 3C lattice constants slightly deviated from those of the ideal cubic structure. In particular, the measured 3C heterofilm lattice is slightly compressed along the in-plane direction and slightly elongated along the out-of-plane direction. 3) 3C films grown by step-free surface heteroepitaxy exhibited narrower diffraction peak widths (17 – 25 arcsec FWHM) and no measurable rotational misorientation with the substrate compared to poorer films we grew on step-free mesas with improper nucleation technique. These results were obtained even for measurements of a 0.4 mm x 0.4 mm SF-free 3C-SiC mesa that was physically isolated (within the X-ray spot) from faulty 3C-SiC elsewhere on the sample (such as 3C-SiC with SF's and DBP's that grew in trench regions and on nearby mesas with screw dislocations). As better described by [11] at this conference, the X-ray results clearly indicate both in-plane and out-of-plane mismatch for the 3C/4H mesa heterostructure.

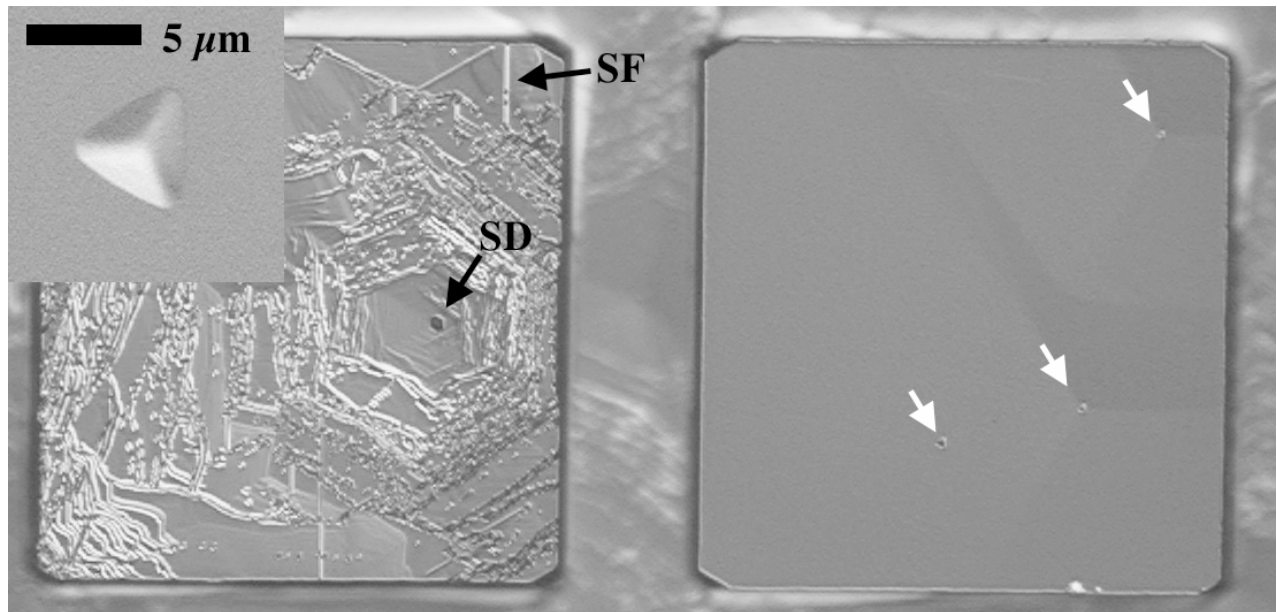
## KOH Etching

Defect-preferential etching is more capable of revealing crystal defects that intersect a film surface at a point (such as a screw dislocation) than thermal oxidation. A piece of Sample C described in [4] was selected to compare oxidation defect mapping with molten KOH defect mapping. Consistent with previous observations, oxidized mesas with screw dislocations exhibited 4H-SiC growth hillocks surrounded by 3C-SiC observed to contain numerous DPB and SF defects [8,13]. The majority of thermal oxides grown on 3C-SiC properly nucleated on screw-dislocation free mesas revealed no DPB's and no SF's.

After stripping the oxide, the sample was etched in molten KOH at 500 °C for 70 sec. As seen in Fig. 2, the KOH etching delineated 4H-SiC screw dislocations (as hexagonal-shaped pits), and all DBP's and SF's previously revealed by thermal oxidation. However, the KOH etch also delineated isolated triangular-shaped etch pits, such as the one shown in the Fig. 2 inset, that were not detected by thermal oxidation. White arrows in Fig. 2 denote the locations of all three such etch pits found on a 200  $\mu\text{m}$  x 200  $\mu\text{m}$  SF-free 3C-SiC mesa. Most 200  $\mu\text{m}$  x 200  $\mu\text{m}$  mesas free of DBP and SF defects typically exhibited 1 to 5 of these isolated triangular etch pits. The etch pit shape and orientation was consistent within any DPB-free mesa, with equilateral etch pit sides (Fig. 2 inset) aligned with three  $\langle 1100 \rangle$  substrate directions. Mesas that contained SF and DPB defects typically exhibited at least an order of magnitude higher triangular etch pit density than SF-free mesas. Further studies are required to ascertain origin of the KOH-revealed triangular pits.

## Summary

Despite the presence of isolated triangular etch pits, the total etch pit density observed on the SF-free 3C heteroepilayers is less than the total etch pit density reported for a 4H-SiC homoepilayer [14]. HRXRD measurements of these films indicate that atoms in the 3C-SiC heterofilm are not all in perfect lateral registration with the 4H-SiC (or 6H-SiC) substrate atoms (i.e., the 3C film is not



**Fig. 2:** 200µm x 200µm mesas following 3C growth and molten KOH etching. Mesa on left is defective (mixed polytype with SF's and hundreds of triangular etch pits in 3C region) due to screw dislocation (SD). 3C mesa on right has three triangular etch pits (inset shows enlarged pit).

fully pseudomorphic). These results are consistent with our proposed model in which remarkably benign partial lattice mismatch strain relief takes place during heterofilm growth [4]. However, further studies are necessary to more definitively understand the observed growth and defect formation behavior.

## References

- [1] J. Wan, M. Capano, M. Melloch, J. Cooper, Jr.: Electronic Mat. Conf. Late Paper F6 (2002).
- [2] G. Gao, J. Sterner and H. Morkoc: IEEE Trans. Electron Devices, Vol. 41, (1994), p. 1092.
- [3] H. Nagasawa, T. Kawahara and K. Yagi: Mater. Sci. Forum Vol. 389-393 (2002), p. 319.
- [4] P. Neudeck, et al: Mater. Sci. Forum Vol. 389-393 (2002), p. 311.
- [5] J. A. Powell et al.: Appl. Phys. Lett. Vol. 77 (2000), p. 1449.
- [6] H. Matsunami: Encyclopedia of Materials Vol. 2 (Elsevier, Amsterdam 2001) p. 1192.
- [7] J. A. Powell, D. Larkin, P. Neudeck and L. Matus: US Patent no. 5,915,194 (1999).
- [8] M. Dudley et al: Mater. Sci. Forum Vol. 389-393 (2002), p. 391.
- [9] J. A. Powell et al: Appl. Phys. Lett. Vol. 59 (1991), p. 183.
- [10] J. Liu, et al: Microscopy & Microanalysis Conf. Paper 419 (2002).
- [11] M. Dudley, X. Huang, W. Vetter and P. Neudeck: this conference Paper We1-01 (2002).
- [12] X. Huang, M. Dudley, P. Neudeck and J. A. Powell: manuscript in preparation (2002).
- [13] M. Dudley, W. Vetter and P. Neudeck: J. Crystal Growth Vol. 240 (2002), p. 22.
- [14] S. Ha, et al: Mater. Sci. Forum Vol. 389-393 (2002) p. 443.

## Acknowledgments

R. Okojie, G. Beheim, L. Matus, L. Keys, B. Osborn, E. Benavage and J. Heisler at NASA Glenn Research Center, and T. Kuhr at Carnegie Mellon University. Funding from NASA Glenn Research Center, US Office of Naval Research and US Department of Energy.